Potentialities of the IET technique for measuring the elastic modulus

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I. INTRODUCTION

Different experimental techniques are used to characterize the mechanical properties of materials, which provide a valuable contribution, not only in the experimental characterization but in the quality control of the production processes. Among the most important parameters that characterize the mechanical properties of materials is the elastic modulus (*E*). It is very useful not only in the understanding of mechanical behavior, but also, it is the basis for calculations and simulations on the stability of the materials, allowing to estimate its behavior and life time under certain stress conditions. The modulus *E* of metals is commonly well known and, in general, it does not depend strongly on the environmental conditions, such as humidity or temperature, for a wide range around room temperature [1]. The situation is very different with polymeric materials, since this parameter depends largely on environmental conditions in a much narrower temperature range. This fact generates a question that must be addressed, and it becomes necessary to study the influence and effects of, for example, the temperature and the water absorption on the mechanical properties, since this type of analysis is useful to estimate the degradation of the polymers quality [2]. The measurement of *E* is indirect and is carried out by methods that can be static or dynamic [3]. Nowadays, the impulse excitation technique (IET) is very used for

having the advantage of being non-destructive and sensitive. Although this method is still not well established, to date there are several experiences reported in the literature [4-10].

The IET technique consists of exciting the natural frequencies of a sample by means of a punctual impact on one of its faces. There are different vibration modes, and in this work, emphasis is placed on the flexural mode of a rectangular section bar and longitudinal mode of a cylindrical section bar. In the first case, the sample is placed on supports in such a way that it oscillates in flexion at natural frequencies. In the second mode, the sample is supported at half the length and is excited by an impact on one side.

For a rectangular section bar in flexural mode, the modulus *E* is given by:

$$E = 0.9465\left(\frac{mf^2}{b}\right)\left(\frac{L^3}{t^3}\right)T \qquad (1)$$

where *f* is the fundamental frequency, *m* is the mass of the sample, *b*, *t* and *L* are the width, thickness and length of the sample, respectively, and *T* is a correction factor and its value is obtained from the literature [1, 11].

An estimation of the frequencies of the harmonics (f_n) respect to the fundamental frequency can be done through the relationship [12]:

$$\frac{f}{9} = \frac{f_n}{(2n+1)^2} \qquad (2)$$

where *n* is the harmonic number (*n*=1 is the fundamental frequency).

For longitudinal vibration in cylindrical section bars with a diameter *D*, the modulus *E* can be obtained as:

$$E = \frac{1}{K_n}\frac{4m}{\pi D^2 L}\left(\frac{2L}{n}\right)^2 f^2 \qquad (3)$$

where K_n is a correction factor. This factor can be determined by different approximations [11]. In the present work, K_n is obtained by fitting the reported data to a fifth grade polynomial function for a Poisson ratio of 0.35. In this case, the fit is a function of D/λ, being λ the wavelength or $2L/n$. More details are given in ref. [9].

In the present work, results obtained by IET in different materials and conditions are presented, showing the potentialities of this technique for its application in the academic and industrial fields. The equipment used for the experimental determinations is a low-cost device that has been specifically developed by the authors [13].

II. EXPERIMENTAL

The IET device for the flexural measurements consists of two sharp supports on which the sample is placed. The supports are located at a distance of 0.224 *L* from the ends of the sample. The vibration of the bar is produced by the impact of a small ball in the center of the bar, and the vibration is recorded by means of an electret microphone attached to an amplifier circuit that allows to store the signals in a personal computer.

For the longitudinal measurements, the cylindrical samples of circular section were supported at half of its length and the impact of the ball is produced at one end, while the microphone is at the opposite side. Details of the experimental configuration can be found in the references [5, 8-10].

The frequency spectrum for each sample was obtained by means of an analysis of Fast Fourier Transform FFT from the oscillations recorded. From this spectrum, the fundamental frequency and the harmonics were obtained (see Figure 1).

Using the IET device, the *E* value in samples of pure aluminum (99.8% of purity), electrolytic copper, the commercial polyacetal resin Derlin®, the commercial polyamide Grilon®, an epoxy polymer and an epoxy matrix filled with aluminum particles composite were determined.

The electrolytic Cu bar was previously thermally treated (annealing at 860 °C for 2 h followed by slow cooling in the furnace).

The epoxy resin (DGEBA) was cured with a hardener (MTHPA anhydride) and an accelerant (tertiary amine) at 120 °C for 14 h, after a slowing heating at 0.8 °C/min.

The particulate epoxy matrix composite was loaded with 15 $\%_v$ aluminum particles with a particle size of 106-125 μm diameter. More details about the fabrication in ref. [5, 13].

All the samples were machined in a milling cutter and, finally, polished with sandpaper.

Measurements at different temperatures

To perform measurements at different temperatures, a heating system was added to the IET device. An electronic temperature controller was used to make a gradual heating of the samples. The temperature was measured with a type K thermocouple placed as close as possible to the sample but without direct contact with it. The temperature increment was carried out in steps, allowing, in each of these, that the temperature stabilizes reaching the thermal equilibrium. In this way, the *E* modulus was obtained as a function of temperature.

In order to have reference values and to evaluate the efficiency of the device, a temperature sweep was performed for the aluminum sample from room temperature up to around 110°C. The values of *E* were obtained from the resonance frequencies using the procedure explained above.

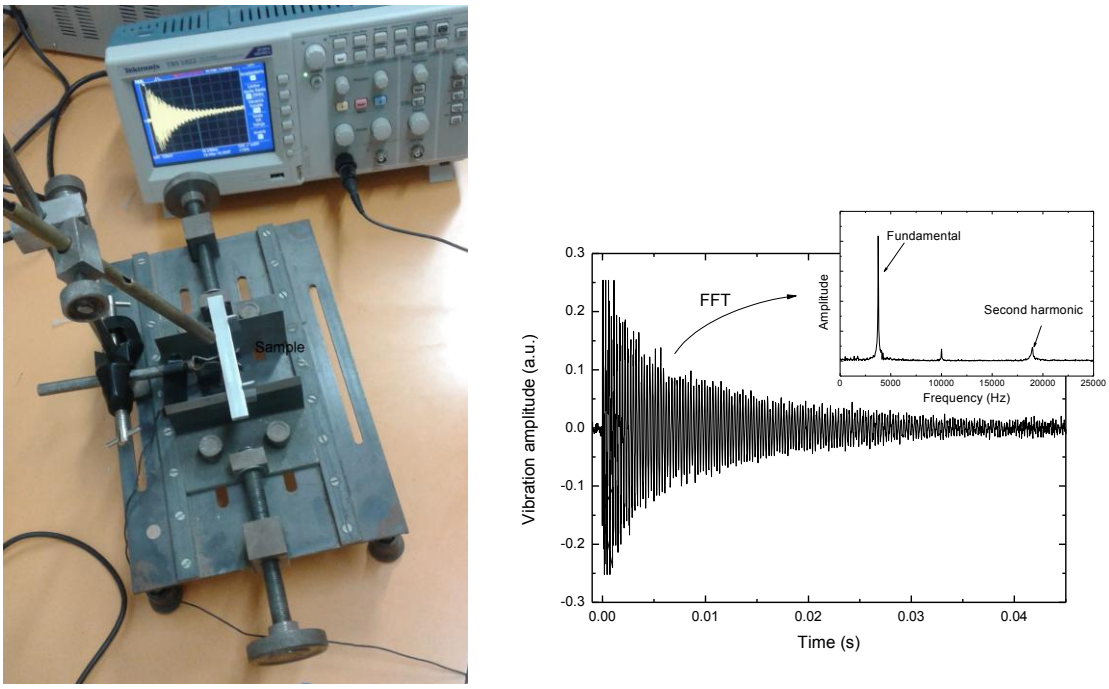

Figure 1. Experimental device. Typical spectrum obtained using a flexural mode and the resultant of the decomposition in frequencies using Fast Fourier Transform.

III. RESULTS AND DISCUSSION

In Figure 2 are presented as an example the spectra of the frequencies present in a vibration in flexural mode, obtained by applying the Fourier transform to the oscillations of copper, epoxy, Grilon and Derlin samples. In all cases, the presence of a sharp peak with a well defined maximum is observed, which is precisely the fundamental resonance frequency (f) of the studied sample. It is also observed that for each sample there is a harmonic (f_3) of considerably smaller amplitude. The ratio f/f_3 was calculated and the obtained values are presented in the fourth column in Table 1. It can be appreciated that f/f_3 is approximately 0.2, which is close to the expected value obtained from the ratio between the third harmonic and the fundamental in equation (2) of 0.18. Also, from the obtained results it is concluded that the second vibration harmonic (n=2) is not detected, which can be attributed to the fact that this harmonic has a vibration node at half the length of the bar in flexural mode. In the present work, the excitation point of the vibration produced by the impact of the ball corresponds to the half of the length of the bar. For this reason, the vibration mode with a node at half the length of the bar (n=2) in flexural mode can not be detected.

Bearing in mind the amplitude, width of the main peak and the signal/noise ratio, it is concluded that with the Cu sample a better signal is obtained. By comparing the spectra of the polymer samples, a certain similarity is observed, although the peak of the fundamental frequency for the epoxy sample exhibits greater amplitude. It is observed that the signal/noise ratio is high enough to analyze the spectra without difficulty in all cases studied, although the Grilon sample has a greater noise than in the other specimens. This greater noise could be attributed to the fact that this polymer is thermoplastic and amorphous, and produces more energy dissipation and the vibration decays faster. This causes the sinusoidal signal is shorter and external signals produce interference. Meanwhile, Derlin has a large percentage of crystallinity, and epoxy has a rigid cross-linked structure. It has also been observed that the signal/noise ratio can decrease when some external parameter varies, such as an increase in temperature or water absorption. The variation of the

oscillation with temperature will be analyzed in following sections, while the variation of *E* due to water absorption was studied previously [8].

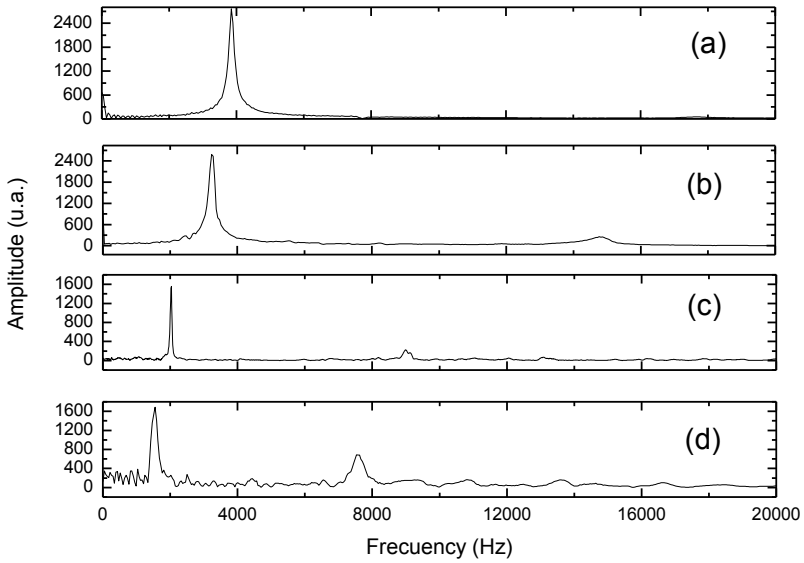

Figure 2. Spectra of frequencies present in a vibration, obtained by applying the Fourier transform to the oscillations of the samples in flexural mode: (a) copper, (b) epoxy, (c) Derlin and (d) Grilon.

Sample	***f*** **(s^{-1})**	***f_3*** **(s^{-1})**	***f/f_3***	***E*** **(GPa)**
Copper	3800.6±0.6	17802.5±2.7	0.21	126±3
Epoxy	3240.1±4.6	14724.1±13.0	0.22	3.86±0.15
Derlin	2025.4±0.8	9022.7±3.3	0.22	3.28±0.12
Grilon	1536.4±4.9	7586.3±5.4	0.20	2.15±0.14

Table 1. Fundamental and secondary frequencies for each studied sample. The ratio f/f_3 obtained for each of them is presented, as well as the value of *E* at room temperature.

Using the values obtained from the fundamental frequencies and applying equation (1), the modulus *E* was determined for each sample and the values are presented in Table 1. They are close to the values reported in the literature [14, 15], so it is concluded that the measurement system exhibits a satisfactory response.

Measurement of modulus E at different temperatures

With the aim of exploring the scope of the IET technique in the study of materials at different temperatures and obtain information about the variation that occurs in the modulus, measurements at different temperatures were carried out. An aluminum sample was measured in a temperature run from room temperature to approximately 110°C. The results were reported in [16], observing a decrease of *E* for higher temperatures with an approximately linear behavior. An adjustment by means of a linear function allowed to obtain a value for the slope of 0.049±0.016 GPa/K and for the ordinate to the origin of 71 ± 1 GPa.

Taking the previous measurements as reference, the same procedure was applied to study the variation of *E* with the temperature in polymeric samples [13]. The obtained results for the epoxy and an epoxy matrix filled with aluminum particles composite are presented in Figure 3.

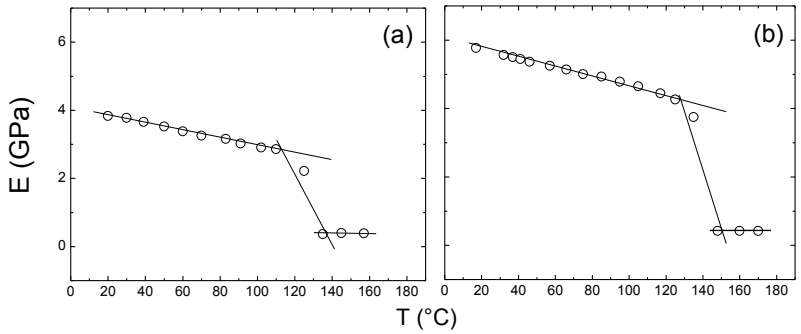

Figure 3. Variation of the modulus *E* with the temperature for the samples: (a) epoxy and (b) epoxy matrix filled with aluminum particles composite.

The epoxy and the epoxy/Al composite exhibit a similar behavior: there is a first region with a linear decreasing of *E* until around 120 ºC, temperature that would correspond to T_g (glass temperature). At higher temperatures, it is found that the resonance frequency has a higher value than in the region of temperatures below T_g. This is because the fundamental frequency has decreased so much that it is masked in the noise and what is being observed is actually the second harmonic. For this reason, and to determinate the values of the modulus at temperatures higher than T_g, a different procedure was done. With the results obtained previously we can expect that the f/f_2 ratio is approximately 0.2. Therefore, knowing

f_2, f can be calculated and then the modulus E. In this way, the values presented in Figure 3 for temperatures higher than T_g were obtained.

The values of the slope (α) and ordinate to the origin (β) corresponding to a linear adjustment of the experimental data at temperatures lower than T_g (glassy state) for each sample are presented in Table 2. In a previous work [17], some of the authors studied the behavior of the modulus E with the temperature in Derlin samples. The obtained results are also included in Table 2.

As is observed in Table 2, the highest variation with the temperature occurs for Derlin, with a slope of approximately twice as that found for the other polymers. On the other side, the composite filled with aluminum particles exhibits a slightly higher variation with the temperature than the epoxy, possibly because the variation of aluminum is greater than that of the epoxy.

	α (GPa/ºC)	β (GPa)	Linear regression coefficient
Epoxy	0.011±0.001	4.09±0.02	0.99
Epoxy/Al composite	0.012±0.002	5.97±0.01	0.99
Derlin [17]	0.025±0.001	4.12±0.12	0.98

Table 2. Slope (α) and ordinate to the origin (β) corresponding to a linear adjustment made to the data of E as a function of the temperature for each sample. The linear regression coefficient that justifies the correct adjustment of the data is also presented.

With the aim of carrying out an evaluation of the results obtained, the glass transition temperature of each sample was measured using the differential scanning calorimetry technique (DSC) in a run at 10 ºC/min [8, 18]. The comparison of the obtained results is presented in Table 3.

Sample	T_g(DSC) (ºC)	T_g(IET) (ºC)	Relative difference of E (%)
Epoxy	119±1	114	90
Epoxy/Al composite	122.2±0.8	119	93

Table 3. Glass transition temperature of each sample, obtained by IET and DSC measurements. The last column presents the change of modulus E when the sample passes from the glassy to the rubber state.

It is necessary to emphasize that the T_g obtained using these two experimental techniques and under different heating conditions are not directly comparable. Moreover, there are usually variations in the T_g values that depend on the type of measurement [19]. However, it can be concluded that in the temperature range used in this work, IET technique shows a good agreement with the results obtained using a technique more specific for the determination of this parameter as is DSC.

In the last column of Table 3, the change of *E* when the sample passes from the glassy to the rubber state is presented. The high percentages evidence the abrupt decrease reported in Figure 3. For temperatures higher than T_g, the vibration is strongly damped and it is difficult to clearly identify a frequency of vibration. It is in accordance with the aforementioned that a lower signal/noise ratio is consistent with a sample with greater energy dissipation.

The behavior of the modulus *E* with the temperature has been also previously studied by the authors in Cu based alloys, specifically in a commercial Cu-2Be alloy and in a shape memory Cu-Al-Be alloy [16]. An approximately linear decreasing of the modulus with the temperature was also found in both samples. Slopes α of 0.034 GPa/°C and 0.047 GPa/°C were determined for the Cu-Be and Cu-Al-Be samples, respectively.

It has been also reported that the measurements of *E* by IET are very sensitive to microstructural changes. In previous works, the authors studied the variations of the modulus *E* with the grain size, the vacancy concentration, and the presence of nanometric and micrometric precipitates [8, 9].

The authors of the present work also carried out experimental measurements by IET in a Cu-Al-Be alloy using the flexural and longitudinal modes in ref. [8]. It was observed that the determination of *E* using the fundamental frequency gives the same information using both modes. This result is of great importance, because it allows to perform measurements in samples with different geometries according to the availability of material, using the most appropriate mode for that geometry. This is particularly important in the industrial field and for measurements in special materials. It is also important to note that this result was obtained in a metallic material, where *E* does not depend on the frequency. To analyze the case of a

material where *E* depends on the frequency, the possible variations of the measured *E* were studied in Grilon samples with different lengths.

Measurement of long rods of a commercial polymer

The vibration frequency in longitudinal mode of a Grilon rod was measured. The length of the rod was reduced by cutting it transversely, so the resonance frequency increments. The modulus *E* was determined from the obtained frequencies for the rods with different lengths. The variation of *E* with the frequency, obtained for the rods with different lengths, is presented in Figure 4. It is observed that the modulus *E*, has an increasing tendency when the frequency increases, although a lot of dispersion can be appreciated. This increasing tendency can not be assumed as an error in the equation (3) that relates the frequency with the modulus. This asseveration was confirmed by a simulation using finite elements method, in which the oscillation frequency was obtained for a material with a given modulus (it was determined from equation (3)). These frequencies coincide with those experimentally measured. Thus, it is inferred that the variation of *E* could be associated to the different response of the material as a function of the frequency. It is known that the dynamic properties of polymers are strongly dependent on frequency, and the IET technique proves to be sensitive to that dependence.

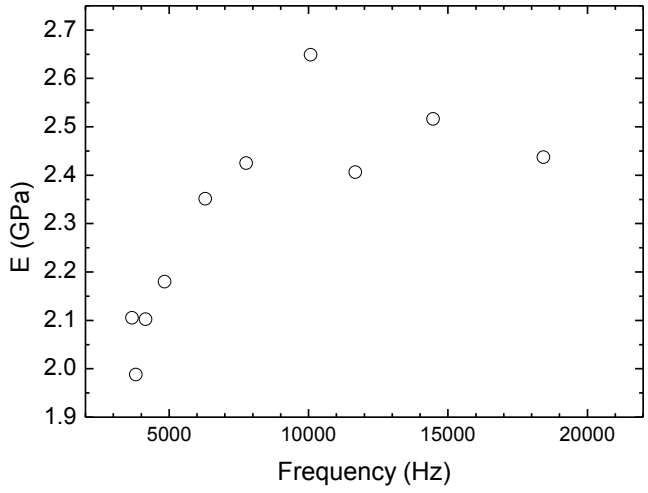

Figure 4. Variation of *E* with the frequency obtained for the rods with different lengths of a commercial Grilon.

IV. CONCLUSION

It has been found that the IET technique is a very a useful tool to obtain reliable results in mechanical properties of polymeric and metallic materials. Information of several polymers with different internal structure has been obtained, observing the response that this device presents when some external parameter is changed, as is the temperature, specifically in the signal/noise ratio of the spectra obtained. It is concluded that a lower signal/noise ratio implies a sample with higher energy dissipation, which depends exclusively on the internal arrangement presented by the material under study.

The values obtained of the modulus *E* are close to those reported in the literature, so it is concluded that the measurement system exhibits a satisfactory response.

The behavior of epoxy and an epoxy matrix filled with aluminum particles with the temperature was studied. The determination of T_g with this technique presents a good agreement with results reported in the literature using specific experimental technique as differential scanning calorimetry.

A particular study for the Grilon polymer was presented, where the sensitivity of the technique to *E* variations due to frequency was evidenced.

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